

## PATENT SPECIFICATION

NO DRAWINGS

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## COMPLETE SPECIFICATION

## Improvements in or relating to the Comminution of Calcium Minerals

We, ENGLISH CLAYS LOVERING POCHIN & COMPANY LIMITED, a British Company, of John Keay House, St. Austell, Cornwall, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to the comminution of calcium minerals and, more particularly, is concerned with the comminution of calcium minerals, such as calcium carbonates, calcium sulphate and calcium silicate, which have a non-sheet crystal structure in contradistinction to minerals, such as the clay minerals, which have a sheet, or laminated, crystal structure.

Chalk whiting, which essentially comprises calcium carbonate, generally to the extent of from 95 to 98% by weight, is a pigment which is used extensively in the manufacture of putty, paints, ceramics and linoleum, and as a filler for rubbers and synthetic resins. In the wet process for the manufacture of chalk whiting, the raw chalk is quarried, ground, classified, filtered, dried, powdered and then packed. In the grinding step it is known to grind the chalk in an aqueous medium in, for example, a ball mill. The conventional methods of grinding the chalk result in the production of a material having a reflectance to violet light (wavelength: 458m $\mu$ ) of about 85% and containing from 25 to 35% by weight of particles smaller than 2 microns equivalent spherical diameter. These properties are quite suitable for its uses as described above, but if the product is to be used in the paper industry, either as a filler pigment or as coating pigment, the brightness is low compared with many English clays used for these purposes. The particle size also is such that its use in

paper coating produces a very matt surface of high ink receptivity. Because of this the calcium carbonates used in paper coating are produced by a chemical precipitation method. These chemically precipitated pigments usually have very much higher reflectance figures of the order of 95% and are often very much finer. Although it has been recognized that it would be desirable to produce a chalk whiting with properties approaching those of the chemically precipitated material, attempts in the past have shown that, by use of methods known heretofore, it is a difficult and expensive procedure.

It has now been found, however, that it is possible to comminute chalk whittings economically in a way such as to make the product finer and to improve its brightness, whilst at the same time maintaining the desirable low adhesive demand (i.e. only a small amount of adhesive, such as starch or casein, is required to hold the chalk whiting onto a sheet of paper), oil absorption and rheological properties of conventional commercially obtainable chalk whittings.

More specifically, in accordance with the present invention there is provided a method of comminuting a calcium mineral having a non-sheet crystal structure, which comprises grinding an aqueous suspension comprising at least 25% by weight of said calcium mineral with a particulate grinding material in the presence of a non-mucilaginous dispersing agent.

In practice, there is provided a suspension of the calcium mineral to be comminuted, e.g. chalk whiting, in water containing a non-mucilaginous dispersing agent. The preferred non-mucilaginous dispersing agent is a water-soluble acrylic polymer, such as Dispex (Trade Mark) or Polysaltz,

(Trade Mark) which is used in quantities of from 0.2 to 0.4% by weight based on the weight of the calcium mineral to be comminuted; other non-mucilaginous dispersing agents, such as the polyphosphates, can of course be used.

In the comminution of the calcium mineral, the concentration of the solid material in the suspension is at least 25% w/w, and most preferably is about 40% w/w; if concentrations below 25% are used, the process becomes economically unacceptable. Generally, concentrations above 50% will not be used since the efficiency of the comminuting process decreases at concentration above this figure.

To the suspension of the calcium mineral to be comminuted there is added a particulate grinding material which is advantageously in the size range of from  $\frac{1}{2}$ " to 100 B.S. mesh and most preferably from -8 to +30 B.S. mesh. Particulate grinding materials which can be used in the present invention include quartz grains, such as Ottawa sand, Molochite which is a proprietary calcined clay product, and various organic materials, for example synthetic resin pellets such as nylon pellets. The grinding material is preferably added to the suspension in an amount such that the ratio by weight of the grinding material to the calcium mineral to be comminuted is in the range of from 2:1 to 5:1, and is preferably of the order of 4:1 although this figure is not critical.

The mixture of suspension and grinding material is agitated either by a tumbling action, as occurs in a conventional ball mill, or by stirring in a stationary vessel for a period of time sufficient to produce the desired size change. The time required is usually of the order of a few hours but varies according to the nature and amounts of materials used and upon the method of agitation used.

The invention is illustrated by the following Example.

#### EXAMPLE

There was prepared a mixture consisting of the following ingredients:

|                                  |          |
|----------------------------------|----------|
| Pulverised whiting               | 1000 g.  |
| Molochite (-10 to +30 B.S. mesh) | 4000 g.  |
| Water                            | 1500 cc. |
| Dispex                           | 2 g.     |

and this mixture was introduced into a ball mill using a Linatex-lined (the word LINATEX is a Trade Mark) cylinder of 6.6 litres capacity rotating at 61 rpm on rollers. Samples were withdrawn from the cylinder for testing after 2, 4, 8 and 16 hours and gave the following results:

| Grinding Time<br>(hrs) | Particle size<br>(% < 2 $\mu$ ) | Reflectance<br>to violet<br>light<br>(458 m $\mu$ ) |    |
|------------------------|---------------------------------|---|----|
| 0                      | 35                              | 85.6%   | 65 |
| 2                      | 54                              | 86.8%   |    |
| 4                      | 70                              | 88.6%   |    |
| 8                      | 86                              | 89.8%   |    |
| 16                     | 97                              | 89.8%   |    |

An attempt was made to repeat the foregoing procedure without the use of Dispex and it was found that no grinding of the chalk whiting occurred due to the high viscosity of the contents of the ball mill. In addition, no change in the reflectance to violet light of the chalk whiting was observed.

#### WHAT WE CLAIM IS:—

1. A method of comminuting a calcium mineral having a non-sheet crystal structure, which comprises grinding an aqueous suspension comprising at least 25% by weight of said calcium mineral with a particulate grinding material in the presence of a non-mucilaginous dispersing agent.

2. A method according to Claim 1, wherein the non-mucilaginous dispersing agent is a water-soluble acrylic polymer and wherein the aqueous suspension contains from 0.2 to 0.4% by weight of said non-mucilaginous dispersing agent based on the weight of the calcium mineral to be comminuted.

3. A method according to Claim 1 or 2, wherein the aqueous suspension contains not more than 50% w/w of the calcium mineral.

4. A method according to Claim 1, 2 or 3, wherein the particulate grinding material consists of particles in the size range of from  $\frac{1}{2}$ " to 100 B.S. mesh.

5. A method according to Claim 4, wherein the weight ratio of the grinding material to the calcium mineral to be comminuted is in the range of from 2:1 to 5:1.

6. A method according to any one of Claims 1 to 5, wherein the calcium mineral is a chalk.

7. A method according to Claim 1, substantially as described in the foregoing Example.

8. A comminuted calcium mineral whenever prepared by the method claimed in any one of the preceding claims.

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